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Effect of Laser Surface Treatment versus Acid Etching on the Microshear Bond Strength of Resin Composite to Dentin: An In Vitro Study

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ABSTRACT— This research seeks to evaluate the effect of laser surface treatment of Er:YAG and Er,Cr:YSGG compared to acid etching on the micro-shear bond strength of nanohybrid flowable composite to dentin tested immediately after bonding and after thermocycling. Forty-eight extracted sound human molars were selected from persons aged between 25-45 years and the reason for extraction was either due to diabetes or periodontal disease. The shear bond strength after bonding of molar tubes using ER-YAG laser was normally distributed with mean of 6.29 standard deviation of 1.21. The micro-shear bond strength of acid etched bonded molar tubes was normally distributed with a mean of 5.08 and a standard deviation of 0.89. A total of 156 selected specimens were divided into three main groups based on surface treatment: ER:YAG laser surface treatment + universal adhesive system; ER;CR:YSGG + universal adhesive system; and acid etching + universal adhesive system (control group). Each group was further divided into four subgroups based on aging period: 24 hours in distilled water, 10,000 thermocycling cycles, 20,000 cycles, and 50,000 cycles. Both conventional acid etching and laser etching showed similar dentin bond strength values. All groups showed reduced bond strength after thermocycling Intragroup comparisons within the Er:YAG, Er:Cr:YSGG, and acid etch groups have shown statistically significant differences between different time periods. Laser etching offers a viable alternative to acid-etch, with fractographic analysis revealing adhesive failure in different surface treatments after just 10000 cycles, contrasting with acid-etched specimens.

KEYWORDS: Laser, Bond strength, Acid etching, Thermocycling.

1. Introduction

Resin composite restorations are increasingly popular due to minimal intervention dentistry and high aesthetic demands. They preserve tooth structure, reinforce it, and offer repair potential. With improved mechanical properties, abrasion resistance, and ease of application, they have become the preferred material for various clinical situations [1], [2]. Bonding to dentin is more challenging than enamel due to its heterogeneous structure, inherent wetness, and permeability. Type 1 collagen is the main component of the dentin matrix. The etch and rinse approach causes discrepancies, hydrolytic degradation, activation of matrix metallo-proteinases, and technique-sensitive collagen collapse, affecting bond durability and resin infiltration [3], [4].

Self-etch adhesives are modified adhesive systems to enhance bonding to dentin and eliminate etching procedures. They condition the dentin surface and facilitate resin infiltration, ensuring deeper demineralization and complete resin infiltration, and enhancing hybrid layer quality and bond durability [5], [6]. A new family of universal or multi-mode dental adhesives has emerged, offering versatility in application methods, and allowing practitioners to choose the most suitable protocol for the cavity being prepared [7- 10].

Laser technology offers a new alternative for cavity preparation, with Er:YAG and Er,Cr:YSGG laser devices performing best with dental hard tissues due to their close proximity to the peak wavelengths. Dentin surfaces irradiated with lasers show microstructural alterations, providing a rougher surface and open dentinal tubules, making them a more suitable substrate for adhesive-bonded restorations. This method can replace phosphoric acid etching, avoiding overly aggressive techniques and collagen collapse [11- 13]. Erbium lasers for tooth-surface conditioning have lower energy densities than cavity preparation, and bonding quality to laser-irradiated dentin may be influenced by laser power output and adhesive system [14], [15].

Acid etching is a technique used to improve bond strength between dentin and restorative materials in dental restorations. However, it is technique-sensitive, requires precise control to avoid over- or under-etching, and cannot effectively remove the smear layer, hindering optimal bonding. Additionally, acid etching may cause demineralization of dentin, potentially causing post-operative sensitivity and compromised long-term restoration [1- 3].

Dentin laser surface treatment using erbium lasers is a promising alternative to acid etching, offering controlled ablation and modification of the dentin surface. This treatment eliminates technique sensitivity and allows for precise surface modification. Erbium laser parameters, such as energy density and pulse duration, can be adjusted to achieve optimal surface roughness without excessive damage to the dentin substrate [4].

Dentin laser surface treatment uses erbium lasers to remove the smear layer, promoting a more intimate bond between dentin and restorative materials. This process exposes the underlying dentin tubules, enhancing hybrid layer formation and bond strength. Erbium lasers operate at a wavelength that minimizes thermal and chemical damage to dentin, absorbing energy predominantly by water. They do not cause demineralization of dentin, eliminating post-operative sensitivity risks and preserving the long-term integrity of the restoration. This controlled surface modification and removal of the smear layer improve bond strength and durability compared to acid etching [7- 10].

The study aimed to assess the effect of erbium laser etching on the bond strength and durability of nanohybrid flowable resin composites with dentin. Despite some studies on bond strengths and immediate bond strengths, there is limited research on the bond durability and bonding stability of adhesives to laser-irradiated dentin. The null hypothesis suggested that surface treatment with erbium lasers would not affect the bond strength or stability of the adhesive bonding system.

2. MATERIALS AND METHODS

Forty-eight extracted sound human molars were selected for this study. Age group was 25-45 years and the reason for extraction was either due to diabetes or periodontal disease. Sample size was estimated based on the results of [16] in which the shear bond strength after bonding of molar tubes using ER-YAG laser was normally distributed with mean of 6.29 standard deviation of 1.21. The micro-shear bond strength of acid etched bonded molar tubes was normally distributed with a mean of 5.08 and a standard deviation of 0.89.

A total of 156 selected specimens were divided into three main groups based on surface treatment: ER:YAG laser surface treatment + universal adhesive system; ER;CR:YSGG + universal adhesive system; and acid etching + universal adhesive system (control group). Each group was further divided into four subgroups based on aging period: 24 hours in distilled water, 10,000 thermocycling cycles, 20,000 cycles, and 50,000 cycles.

The present study involved embedding each tooth in acrylic resin using auto-polymerized resin in a mold, pressing it into the resin, and then checking the surface for superficial dentin. A standard smear layer of exposed dentin surface was created using 600-grit silicon carbide papers, marking a point 1 mm below the dentino-enamel-junction [17].

The laser treatment used for the first group was a 2940 nm Er:YAG laser with settings of 120 mJ, 10 Hz, and 1.2 W for 10 seconds, with water and air spray levels of 6, and a sapphire tip 1.3mm in diameter. The beam was delivered perpendicular to the tooth surface, as shown in Figure 1. For 10 seconds, a 2780 nm Er,Cr:YSGG laser therapy with conditions of 120 mJ, 20 Hz, and 1.5 W was employed for the second group. The water spray flow was 75%, and the air level was 60%. The beam was supplied perpendicular to the tooth surface at a constant working distance using a sapphire tip, as illustrated in Figure 2. The control group didn't receive surface treatment, and the adhesive system will be applied according to the manufacturer's instructions, followed by 15 seconds of acid etching.

A disposable micro-brush was used to apply a universal adhesive to the dentin surface, followed by rubbing for 20 seconds. The adhesive was left uncured to fix the cut microtubes before packing the resin composite into them. A polyethylene micro-tube with a diameter of 0.9 mm and 1mm was mounted on an uncured adhesive. The adhesive was then light-cured for 10 seconds using an LED light-curing unit. The light cure tip was held on the top of the micro-cylinder to standardize the curing distance (2 mm from the adhesive) [18].

The composite syringe was used to insert a flowable resin composite into a tube, which was then light-cured for 20 seconds, as seen in Figure 3. After one hour of storage, the polyethylene microtubes were removed, forming composite microcylinders of 0.9 mm diameter and 1 mm height. The specimens were then left in artificial saliva for 24 hours before testing.

The study tested microshear bond strength after artificial aging using 10,000, 20,000, and 50,000 cycles equivalent to 1, 2, and 5 years in clinical conditions. The water baths had dwell times of 25 seconds, a low-temperature point of 5°C, and a high-temperature point of 55°C. Microshear bond strength was evaluated after 24 hours of storage and thermocycling. Tooth in an acrylic mold with bonded micro-cylinders was secured to a universal testing machine with a 5 KN load cell. The data was recorded using computer software. An orthodontic wire loop was wrapped around the bonded micro-cylinder assembly. A shearing load with tensile force was applied to a micro-cylinder at a slow crosshead speed, resulting in de-bonding along the substrate-adhesive interface, and recorded in Newton.

Hybrid layer assessment was done using a Scanning Electron Microscope (SEM) (Quanta FEG 250, Philips Electron Optics, Eindhoven, Netherlands) at 5000x. The specimens of each group were examined immediately and after the thermocycling of the 10000, 20000, and 50000 cycles. After the microshear test, the specimens were observed using a USB digital microscope with a magnification of x35. Images of the specimens were captured and transferred to a personal computer equipped with Image-Tool software. The purpose was to determine the failure mode pattern, which was categorized as adhesive, cohesive within the composite, or mixed failure types.

3. Statistical analysis

The study used Medcalc software to analyze the data, examining normality using Kolmogrov-Smirnov and Shapiro-Wilk tests. Continuous data showed a normal distribution with a mean and standard deviation. Intergroup comparisons were performed using one-way ANOVA and the tukey post-hoc test, while intragroup comparisons were done using repeated measures ANOVA and the tukey post-hoc test. Categorical data was described using frequency and percentage, with a P value of less than 0.05.

4. RESULTS

Concerning the effect of surface treatment on micro-shear bond strength within each time, intergroup comparison has shown no statistically significant difference within time periods; immediate and after 1, 2, and 5 years of thermocycling, as observed in Figure 4. While Table (1) demonstrated the effect of time on micro-shear bond strength within each surface treatment, it revealed that intragroup comparisons within the Er:YAG, Er:Cr:YSGG, and acid etch groups have shown statistically significant differences between different time periods ($P < 0.001$). Micro-shear bond strength decreased gradually within each surface treatment with time, with the least bond strength after 5 years of thermocycling.

Intergroup comparisons between the surface treatments have shown no statistically significant difference within time periods; immediate and after 1 year ($P = 0.8402$ and $P = 0.2767$), respectively, while after 2 and 5 years of thermocycling, there was a statistically significant difference between surface treatments ($P = 0.0006$ and $P = 0.0002$), respectively, as explained in Figure 5. According to Table (2), there were significant differences in immediate failure modes within different groups, with mixed and cohesive failure modes appearing initially and adhesive failure modes not appearing until 5 years after simulated thermocycling. The same pattern was observed in the Er:Cr:YSGG group, where immediate failure modes were mostly mixed and cohesive, and in the acid etch group, where adhesive failure was earlier.

Morphological analysis of the specimens by scanning electron microscope showed different characteristics according to the surface pre-treatment, as shown below: A) Er:YAG laser irradiation: The eroded dentin treated with Er:YAG laser showed a surface without a smear layer with disrupted tissue and demineralized collagen fibrils. The micrographs show highly irregular dentin with a scaly appearance, suggesting characteristics of ablation, with open dentinal tubules and prominent peritubular dentin, as observed in Figure 6a. B) Er,Cr:YSGG laser irradiation: The eroded dentin samples that were subjected to surface treatment with the Er,Cr:YSGG laser showed an irregular and rough surface, with open dentinal tubules and prominent peritubular dentin without a smear layer, as demonstrated in Figure 6b. Acid etching: The eroded dentin samples which were subjected to acid etching showed a regular surface without a smear layer and with open dentinal tubules, as found in Figure 6c.

Figure (1): Surface treatment using ER: YAG laser beam.

Figure (2): Surface treatment using Er,Cr:YSGG laser beam.

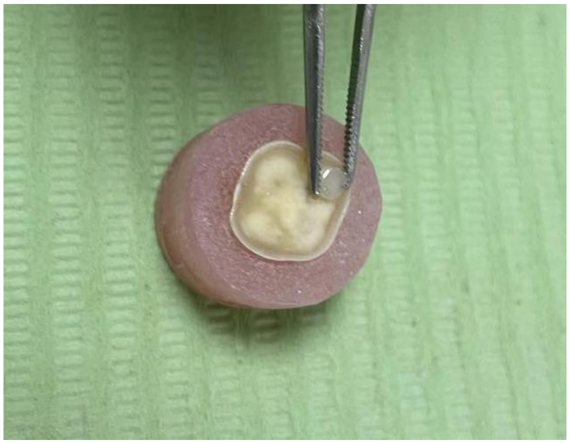

Figure (3): Resin composite micro-cylinder preparation and application.

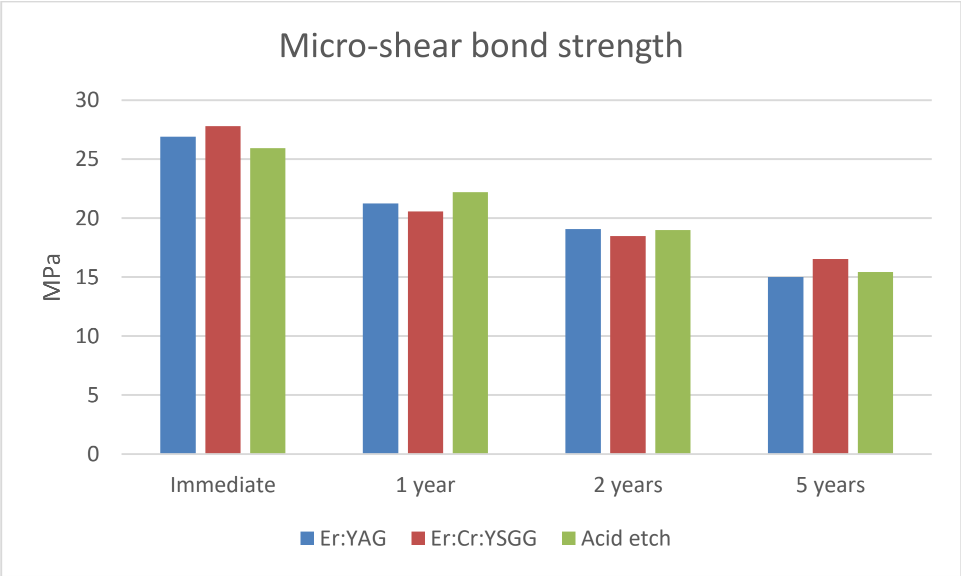

Figure (4): Effect of surface treatment on micro-shear bond strength within each time period.

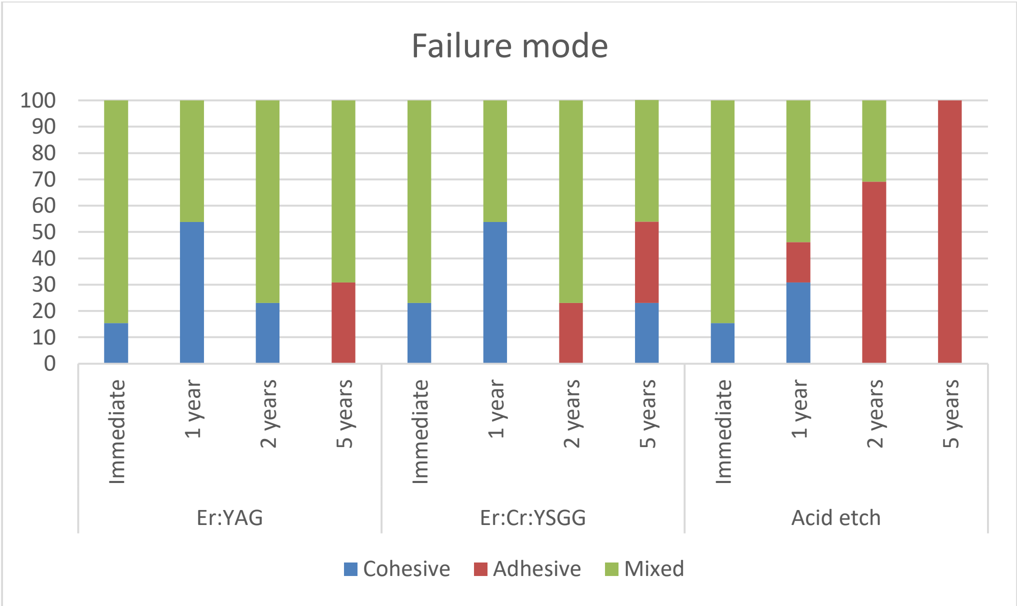

Figure (5): Effect of surface treatment on failure mode within each time period.

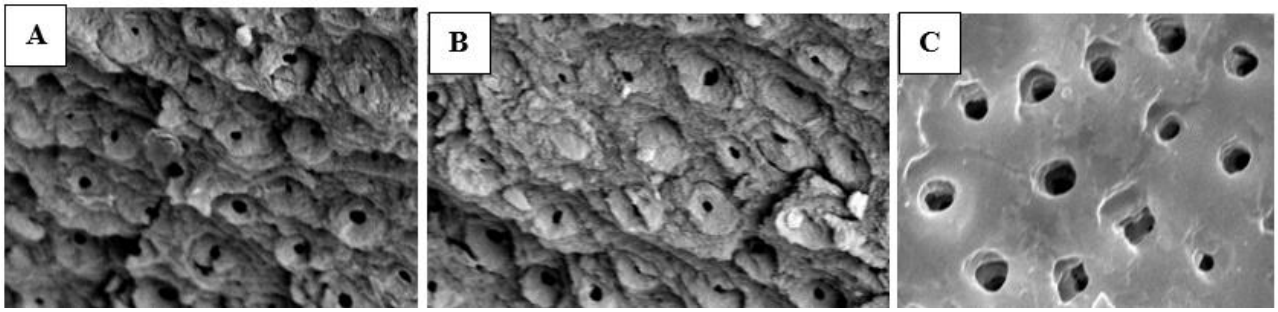

Figure (6): Morphological analysis by scanning electron microscope: A) Er:YAG laser irradiation. B) Er,Cr:YSGG laser irradiation. C) Acid etching.

Table (1): Mean and standard deviation of micro-shear bond strength of surface treatments showing the effect of time within each surface treatment

Intervention / Time	Er:YAG		Er:Cr:YSGG		Acid etch	
	Mean	SD	Mean	SD	Mean	SD
Immediate	26.92^{a}	3.82	27.80^{a}	3.84	25.94^{a}	3.53
1 year	21.24^{b}	1.87	20.56^{b}	1.62	22.19^{b}	1.65
2 years	19.07^{b}	1.52	18.48^{bc}	1.77	19.01^{c}	1.61
5 years	15.02^{c}	2.96	16.56^{c}	2.096	15.45^{d}	1.70
P value	P < 0.001*		P < 0.001*		P < 0.001*	

Means that do not share a letter vertically are significantly different,
* corresponds to statistically significant difference

Table (2): Frequency and percentage of failure mode showing the effect of time within each surface treatment

Time	Er:YAG			Er:Cr:YSGG			Acid etch		
	Cohesive	Adhesive	Mixed	Cohesive	Adhesive	Mixed	Cohesive	Adhesive	Mixed
Immediate	2(15.4%)	0(0%)	11(84.6%)	3(23.1%)	0(0%)	10(76.9%)	2 (15.4%)	0(0%)	11(84.6%)
1 year	7(53.8%)	0(0%)	6(46.2%)	7(53.8%)	0(0%)	6(46.2%)	4(30.8%)	2 (15.4%)	7(53.8%)
2 years	3(23.1%)	0(0%)	10(76.9%)	0(0%)	3(23.1%)	10(76.9%)	0(0%)	9(69.2%)	4(30.8%)
5 years	0(0%)	4(30.8%)	9(69.2%)	3(23.1%)	4(30.8%)	6(46.2%)	0(0%)	13(100%)	0(0%)
P value	P = 0.0011*			P = 0.0097*			P < 0.0001*		

5. DISCUSSION

Over the past 20 years, the bonding of resin-based composite restorations has been developed through continuous advances in dentin bonding technology. Bonding to enamel has proved to be reliable, while bonding to dentin is known to be more difficult due to its heterogeneous, complex structure histologically with inherent wetness [4]. A number of studies have investigated the etching effects of lasers on enamel and dentin surfaces and showed that tooth structures etched with lasers show micro irregularities and no smear layer [19]. Numerous reports have proven the laser etching technique to be effective for bonding. However, very few studies have compared both Er:YAG laser etching and the Er,Cr:YSGG laser etching. Hence, this study was conducted to determine whether Er,Cr:YSGG and Er:YAG laser irradiation may be an alternative method for etching dentin surfaces [16].

In the present study, a universal nano-hybrid highly filled flowable composite was selected since nanotechnology is of great interest in resin composite research. Due to the reduced dimension of the particles and their wide size distribution, an increased filler load can be achieved, reducing the polymerization shrinkage and increasing mechanical properties such as tensile strength, compressive strength and resistance to fracture [20].

Regarding the bond strength, the findings of this study revealed that all three groups showed similar microshear bond strength with no statistically significant difference between them within all time periods (immediate and after 1, 2, and 5 years of thermocycling). This indicates that both Er:YAG and Er,Cr:YSGG laser irradiation may be an alternative method for dentin etching. These findings are in agreement with several studies done by [16], [21- 23] where they also found that Er:YAG and Er;Cr:YSGG laser etching resulted in high microshear bond strength values that were similar to acid etching and that they can be considered a viable option as a surface treatment alternative to acid etching.

In the case of etching with phosphoric acid, smear-layer removal and dentine surface demineralization occur, which results in the formation of resin tags in the open dentinal tubules with the impregnation of the adhesive into the exposed collagen network, creating a well-impregnated hybrid layer, which might explain the improved bond strength [24].

It was found that the dentin surface irradiated with laser showed an absence of a smear layer and a scaly or irregular surface, and the orifices of dentinal tubules were exposed. The absence of a smear layer and exposed tubules might have enhanced the bonding [19]. The laser treatment improved the dentin surface roughness and surface area by opening the dentinal tubules and facilitating the penetration of adhesive into the dentin tubules. Such changes without a smear layer are believed to provide suitable surfaces for strong bonding with composite resin materials, resulting in improved bonding. In contrast, drilling dentin with an air turbine hand-piece leads to the formation of a smear layer on the surface of the dentin, blocking the dentinal tubules as well as interfering with the penetration of the resin adhesive and bonding with restorative materials [21].

Several factors have been speculated to affect the durability of resin-dentin bond over time. The fact that universal adhesives are comprised of a heterogeneous composition that mixes various different components into the same solution (e.g., acidic and non-acidic monomers, solvents, fillers, initiators, and the combination of these factors may have probably decreased the bonding ability to dentin and thus, more liable to degradation [25]. The major cause of degradation is thought to be based on both hydrolysis and enzymatic breakdown of the collagen fibrils and the polymerized resin matrix in the hybrid layer. Degradation was attributed to the hydrophilicity of the adhesive layer which leads to water uptake and plasticization. Water is an indispensable component in these adhesives. Remnant water that cannot be fully removed from the evaporated adhesive due to either decrease in vapor pressure due to the addition of water-soluble resin monomers or the presence of an osmotic gradient provides the hydrolytic fuel for hydrolysis of polymeric resins and enzymatic degradation of collagen fibrils [5].

Endogenous gelatinolytic/collagenolytic enzymes can degrade the collagen structure of the bonded interface over time, accelerating the degradation of the adhesive interface, and this phenomenon has been mainly related to the degradation of hybrid layers created by etch-and-rinse adhesives. The acidity of etchant reduces the pH of the environment activating pro-forms into active forms of MMPs via the cysteine- switch mechanism that exposes the catalytic domain of these enzymes that were blocked by pro-peptides [26]. It is also known that bond-strength reduction and extensive interfacial nanoleakage are more commonly expressed in simplified adhesives compared to multi-step adhesives. In particular, the lack of a hydrophobic bonding resin in one-step universal adhesive formulations has been demonstrated to reduce bond stability over time, because the bonded interfaces behave as semi-permeable membranes allowing the movement of water across them and expediting hydrolytic degradation [27].

Regarding failure mode, the results of this study showed no statistically significant difference between the three groups within time periods; immediate and after 1 year of thermocycling. However, after 2 and 5 years of thermocycling, there was statistically significant difference between the three groups. In accordance with this study, the increased number of adhesive failures seen in the etch and rinse technique was also evident in studies reported by [28- 30] where they also reported that most of the specimens showed adhesive failures. Also, similar findings were found in studies done by [31], [15] where they also reported that after thermocycling, the failure mode in laser-irradiated dentin was mainly mixed and cohesive failures compared to the acid etch group where the predominant failure mode was adhesive failure. This may confirm the fact that laser etching promotes improved bonding due to its tissue ablation that creates a roughened, scaly irregular dentin surface, increasing the surface area for bonding, advocating for proper mechanical interlocking of the adhesive to dentin with no fear of increased depth of demineralization or denuded exposed collagen fibrils that are more prone for degradation [18], [32].

The type of bond failure observed in the present study with the acid etch group being mainly adhesive failure may be attributed to the fact that phosphoric acid decalcifies dentin deeper (up to 3–6 mm) which results in inconsistency in the collagen fibril resin encapsulation due to the high residual water content through the whole depth of demineralization zone and the limited penetration of adhesive resin due to its certain degree of viscosity. Accordingly, this discrepancy between depth of demineralization and depth of monomer infiltration influences hybrid layer formation, leading to a low quality hybridization, in the form of a porous and poorly resin infiltrated collagen mesh, resulting in an adhesive interface that is more vulnerable to biodegradation [29]. The negative effects of acid etching dentin may be the combined result of over-etching, sub-optimal removal of the phosphoric acid; incomplete adhesive infiltration into the denuded collagen network and removal of residual hydroxyapatite from the collagen mesh, which could compromise the potential for chemical adhesion [18].

This study found that laser etching is a promising alternative to acid etching in dental restorative procedures. Acid etching can have negative effects on the dentin surface, weakening the interface between the restoration and the tooth. Laser etching helps form a more stable and durable hybrid layer, resulting in restorations with higher longevity and lower rates of recurring cavities. Additionally, acid etching activates enzymes that degrade collagen in the dentin-resin interface, which can compromise the durability of bonded restorations. Laser treatment does not increase collagen degradation, making it a potentially better option. However, further research is needed to explore the impact of laser etching on dentin and to evaluate the success of these procedures in clinical trials.

6. Conclusion

Under the limitations of this study, it can be concluded that:

- Both conventional acid etching and laser etching showed similar dentin bond strength values, so laser etching can be successfully used as an alternative to the conventional acid-etch.
- Bonds created by acid etching or laser etching are generally incapable of defying aging as a

decrease in bond strength was observed in all groups after thermal aging.

- Although all groups showed reduced bond strength after thermocycling, failure mode by fractographic analysis might help give an idea about the degree and pattern of degradation of each surface treatment showing adhesive failure in acid etched specimens after just 10000 cycles where as in Er,Cr:YSGG adhesive failure was evident at 20000 cycle and in Er:YAG adhesive failure was evident at 50000 cycles.

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Conflicts of interest
The authors declare that they have no competing interests.

Authors' contribution
Conceptualization, motivation, writing-original review, editing manuscript. All authors approved the final draft of the manuscript. All authors participated in the study design, performed the experiments, collected and analyzed the data, drafted the manuscript, and confirmed the authenticity of all the raw data. All authors read and approved the final manuscript.

Ethical policy and institutional review board statement
The study was carried out in Ain Shams University and approved by the research Ethics Committee, Faculty of Dentistry, Ain Shams university, Cairo, Egypt (FDASU-REC) with approval number FDASU-RecEM011931

Patient declaration of consent
Informed consents were taken from the participants prior to enroll them in the study.

Data availability statement
The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

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